A STUDY OF THE PACKING CHARACTERISTICS OF FIBERGLAS PACKING AS ADAPTED TO COUNTERCURRENT LIQUID-LIQUID EXTRACTION

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Emmett Herschel Covington

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ABSTRACT

This investigation was made to compare the performance characteristics of Fiberglas and one-half inch Raschig Ring tower packing in a 5.5 inch diameter liquid-liquid extraction column. The system used was toluene, benzoic acid, and water. Water flows ranged from 30.5 to 74.4 cubic feet per hour per square foot column area for toluene flows of 11.6 to 21.3. A. P. Colburn's H.T.U. method of correlation was used to correlate these data. The separation efficiency of the Fiberglas packing was found inferior to the Raschig Rings, however, the former allowed much higher flow rates before the column flooded.

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INTRODUCTION

As has been the case with many other chemical unit operations the industrial use of liquid-liquid extraction has proceeded the basic theoretical principles underlying the operation. Large scale operations involving the use of liquid-liquid extraction have been in use for some years. However, development of new, and improvement of existing, equipment have emphasized the need for study of the performance of the various types of columns and systems. To date design work on this operation has been greatly retarded by the lack of published performance data. In recent years much progress has been made both in the practical and in the theoretical phases of this operation by such men as Strang, Hunter, and Nash[1]; Elgin[2]; Colburn[3]; and Row, Koffolt and Withrow[4]. Colburn in his work has pointed out the great usefullness of the H.T.U. (height of a transfer unit) and has developed a method of correlation of data using overall H.T.U.'s that is

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1 Strang, L. C., Hunter, T. C. and Nash, A. W.; Industrial Engineering Chemistry, 29; 278, (1937)

2 Elgin, J. C. "Teaching of Chemical Engineering", American Institute of Chemical Engineers, New York (1940)

3 Colburn, A. P., Transactions American Institute of Chemical Engineers, 35, 211 (1939)

4 Row, S. B., Koffolt, J. H., and Withrow, J.R.; Transactions American Institute of Chemical Engineers, 37, 559 (1941)

quite simple and extremely useful. Colburn's equation for data correlation will be used in this thesis.

For the last five years Owens-Corning Fiberglas Corporation has produced a tower packing for use in absorption, distillation, and extraction operations that has been used quite extensively in the chemical industries; however, to date no performance data on this packing in the liquid-liquid-field has been published. Design work with this packing is still in the hit and miss stage as a consequence. The purpose of this thesis is to supply and correlate some experimental data on the performance of this packing and to compare the packing with other packings whose performance is known. The chemical system used; namely, toluene-benzoic acid-water is the same as used by Row, Koffolt, and Withrow[5] in their investigations of various other packing materials. This system was chosen in view of using the published equilibrium data and thereby eliminating the necessity of equilibrium determinations in this problem. However, attempted check points indicated the need of a redetermination of the equilibrium data. Experimental equilibrium data was taken and used throughout this work.

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5 Row, S. B., Koffolt, J. H., and Withrow, J. R.; Transactions American Institute of Chemical Engineers, 37, 559 (1941)

EQUIPMENT

A diagrammatic sketch of the liquid-liquid extraction unit is shown in Figure 1, and a photograph showing the overall view of the unit is illustrated by Figures 12 and 13 in Appendix III.

The Extraction Column - The column itself consisted of five sections of "Pyrex" glass pipe, the average inside diameter of which was 5.50 inches, with an overall height of 101.75 inches. The cross section area was 0.1655 square feet, the effective height of the column for all runs except series "B" was 72 inches giving an effective volume of 0.990 cubic feet. For series "B" an effective height of 82 inches was used. The change in the packed height was made to give a longer calming section at the base of the column.

The column was supported by a tower frame made of one and one-quarter inch angle iron. The glass pipe sections were joined together with metal flanges which also carried the column weight to the frame. The bottom portion of the column consisted of three permanently placed sections with the other two sections comprising the top portion. The bottom and top sections were joined by a bolted flange arrangement. The bottom and top flanges carried all pipe connections to the column. The glass sections were sealed into the flanges with a mixture of litharge and glycerine, made up to a consistency of that of a fairly stiff putty. Saureisin No. 31 was used as a final topping for this seal and to repair any

minor leaks in the sealing.

Tanks - The tanks used in the extraction unit had the following capacities:

Water Supply	- 300 gallons
Toluene Supply	- 200 gallons
Toluene Receiving	- 200 gallons
Constant Head	- 5 gallons each

All tanks were of metal construction manufactured by the Bridwell Boiler Works, Atlanta, Georgia. The tanks used in the toluene system were of necessity unpainted.

Pumps - The toluene supply pump was a centrifugal, three to four gallon per minute pump driven by a one-fourth horsepower explosion proof motor. The water pump was centrifugal, four gallon per minute pump driven by a one-fourth horsepower motor.

Water Supply - Atlanta city water was brought into the water supply tank where the pH was adjusted with hydrochloric acid to a neutral methyl orange end-point. The water temperature was adjusted by the use of open steam to within one-half degree of the toluene temperature. These temperatures varied from 20 to 25°C. The water was then pumped from the water supply tank to a constant head tank, the overflow from the constant head tank returning to the supply tank. From the constant head tank the water flowed by gravity through a calibrated orifice and a throttling valve to the water distributor. The orifice was connected to a manometer which gave a direct reading of the orifice differential pressure. The distributor

was a two inch pipe with the opening covered with a plate perforated with one-fourth inch openings. The water from the bottom of the tank was taken off through an adjustable leg mounted on pivot joints, which could be raised or lowered as required to maintain the interface in the column at any desired point. The water from the leg flowed to the sewer.

Toluene Supply - The toluene was stored in four 55 gallon drums, so connected that the toluene could be pumped from either or all drums and returned to any desired drum or drums. The toluene was taken from storage by the supply pump and received in the supply tank. The same pump was used to pump the toluene to the constant head tank. The overflow from the constant head tank was returned to the supply tank. From the constant head tank the toluene flowed by gravity through a calibrated orifice and a throttling valve to the toluene distributor in the lower part of the column. The orifice was connected to a manometer. The distributor consisted of a two inch pipe cap with 17 holes of 50/1000 inch diameter. The distributor was placed so as to give an eight inch calming section at the bottom of the column. The toluene was taken from the top of the column and flowed by gravity to the toluene receiving tank. Here the toluene was brought back up to the proper concentration with benzoic acid and pumped either to the supply tank for another run or to storage.

Manometers - The manometers were the conventional inverted "U" type using acetylene tetrabromide (specific gravity

of 2.964 $\frac{24^{\circ}}{4}$) as the heavier fluid. In the toluene manometer a leg of water was maintained between the heavier liquid and the toluene to facilitate using natural rubber tubing as connectors between the saran leads and the glass manometer. An arrangement of stop cocks was used to zero the manometer before a run. The manometers were calibrated with rotameters using constant viscosity floats. These readings were checked using the direct weighing method. The compiled and plotted calibration data was included in the Appendix.

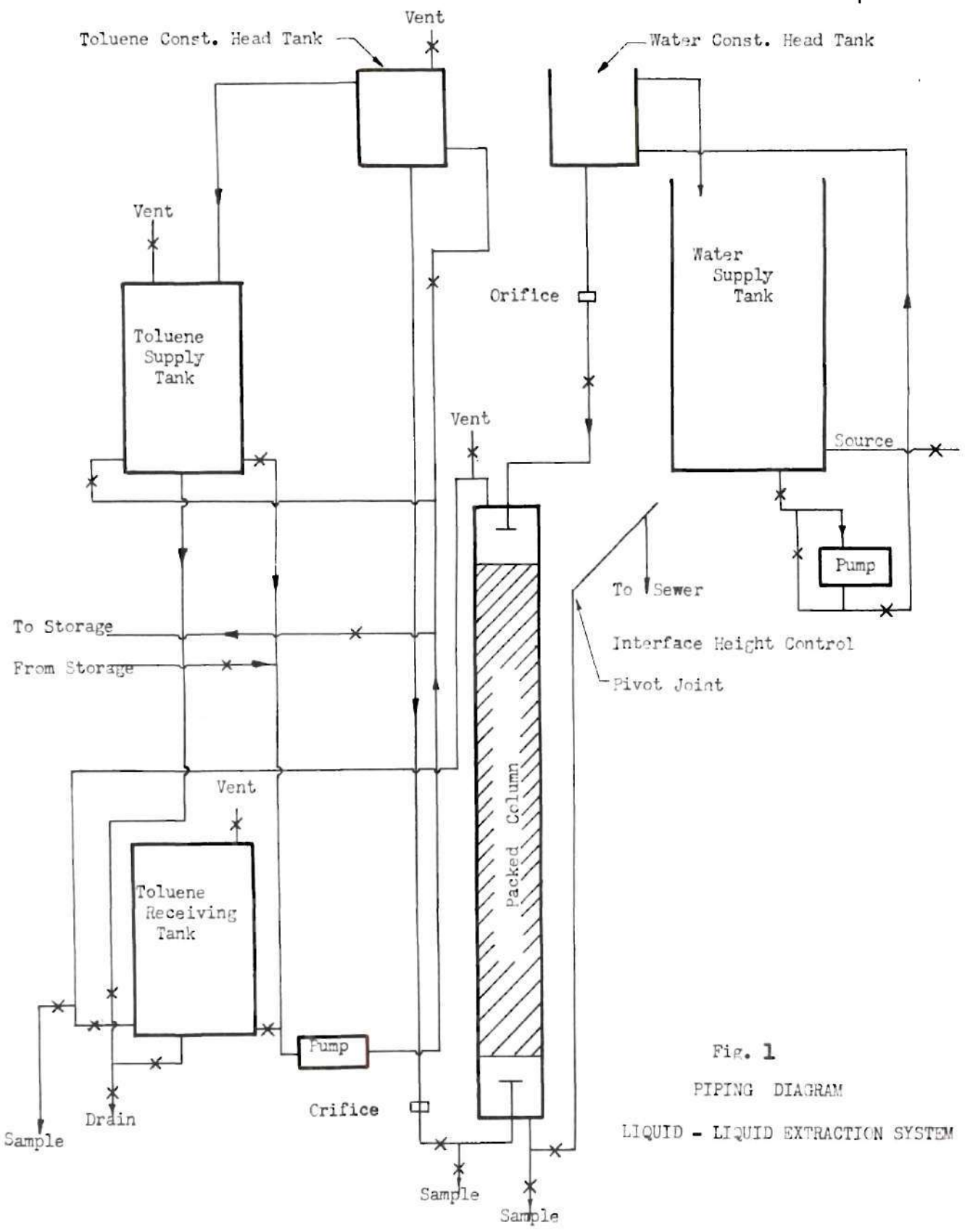

Fig. 1

PIPING DIAGRAM

LIQUID - LIQUID EXTRACTION SYSTEM

MATERIALS

Fiberglas*- The Owens-Corning Fiberglas Corporation, Toledo, Ohio, furnished their tower packing free of charge for use in this work. The packing was received in packs 20" by 20" by 1". The individual glass fibers of which the tower packing is made were arranged in a jack-straw fashion and bonded together with a water-soluble binder to give them sufficient rigidity for easy handling during application. Tests on the packing revealed that the binder constituted 3.56% of the total weight of the packing. The individual fiber had an average diameter of 0.0080 inches. The fibrous glass was charged into the column with the use of a cylinder of adjustable diameter made by rolling a piece of one-eighth inch sheet brass. The cylinder was twenty five inches high and had a maximum diameter of seven inches. Chords of the circular cross section of the inside of the column were measured. Pads of the fiberglas were then cut to stand edgewise so that one dimension corresponded to the dimension of the chord and the other to the original 20 inches of the glass pad. The cut pads were charged to the adjustable cylinder, those cut for the outermost chords were charged first. After several pads had been placed in the cylinder, the inner pads were inserted

*Fiberglas is the trade name (Reg. U. S. Pat. Off.) of glass manufactured in any fiber or filament form by the Owens-Corning Fiberglas Corporation. Fiberglas in this paper refers only to the tower packing.

by sandwiching the pad between two sheets of stiff paperboard and pushing them down into place in the cylinder and withdrawing the boards. This prevented adjacent pads from snagging and tearing the pad being inserted. This procedure was continued until the desired packing density was slightly exceeded. A one-half inch metal screen supported on the outside by a metal ring with three metal upright support rods attached to the metal ring was then inserted in the adjustable cylinder. Another metal ring and screen was inserted from the opposite end and attached to the free ends of the support rods. The outside diameter of the adjustable cylinder was then reduced to slightly less than the inside diameter of the column by use of adjusting straps on the outside of the cylinder. The previously tared cylinder was then weighed to give the actual packing density. The packing was then charged into the column by inserting one end of the cylinder approximately three inches into the column and pushing the Fiberglas from the cylinder into the column. This procedure was repeated until the desired packed height was reached. The bottom and top portions of the column were packed separately and then joined by a bolted flange arrangement. The lower section of the bottom portion was left empty for use as a calming section and to house the dispersed phase distributor. The upper half of the topmost section was also left unpacked to provide space for the continuous phase distributor and an upper calming section. After the column was completely packed and assembled, water was introduced and allowed to stand for eight hours and drained. This procedure

was repeated two times to render the fiberglas completely free of the water-soluble binder.

Raschig Rings - One-half inch, unglazed ceramic, Raschig Rings purchased from the Maurice A. Knight Company, Akron, Ohio, were used. The column was packed with these rings by filling the tower with water and dumping in the packing material from the top of the tower, thereby producing a random arrangement of the rings in the column.

A summary of the packing data is given below in Table I.

TABLE I: Summary of Packing Data

	Fiberglas	Raschig Rings
% Free Space	97.3	53.8
Total Units Charged (No.)	--	7,732
Total Weight Charged (Lbs.)	40.6	64.1
Packing Density (Lbs./cu. ft.)	4.0	64.8

$$\% \text{ Free Space} = \frac{\text{Cu.Ft. water drained from top to bottom level of packing}}{\text{Effective column volume in cubic feet}} \times 100$$

Toluene - The toluene used in this work was a nitration grade purity and was furnished without cost by the Humble Oil & Refining Company, Houston, Texas. The toluene had an A.S.T.M. boiling point of 111.8°C. ± 0.5°C. and a specific gravity of $0.868\ \frac{15}{4}$.

Water - Atlanta, Georgia, tap water was used throughout this work.

Benzoic Acid - A chemically pure grade of benzoic acid purchased from Merck Chemical Company was used.

EXPERIMENTAL

Equilibrium Distribution - Two references in the literature were found on equilibrium distribution data for the system used; the first by Appel and Elgin[6], and the second, by Row, Koffolt, and Withrow[7]. However, preliminary investigations could not check either set of data as closely as desired. The discrepancies were believed caused by the differences in the particular materials used in the investigations. The former of the above investigators used chemically pure toluene and distilled water, the latter of the investigators used Dow Chemical Company's U.S.P. grade benzoic acid, Barrett's nitration grade toluene and Columbus, Ohio, water; while in this work Merck Chemical Company's C.P. grade benzoic acid, Humble Oil & Refining Company's nitration grade toluene, and Atlanta, Georgia, tap water were used. As a result of these differences, it was necessary to determine the equilibrium distribution for the system used by the writer. Determinations were made at 20°C and 25°C. The procedure used for these determinations was the same as used by the above investigators and is summarized as follows:

Samples of benzoic acid were weighed into tared 250 milliliter flasks. One hundred milliliters of toluene and one hundred milliliters of tap water (neutralized to a methyl orange

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6 Appel, F. J., and Elgin, J. C.; Industrial Engineering Chemistry, 29, 451 (1937)

7 Row, S. B., Koffolt, J. H. and Withrow, J. R.; Transactions American Institute of Chemical Engineers, 37, 559 (1941)

end point with hydrochloric acid) were introduced into the flask. The samples so prepared were corked and sealed with petroleum wax and placed in a water bath which could be held constant at the desired temperature. The samples were allowed to stand for ten hours with vigorous shaking every fifteen minutes of the first three hours and at one-half hour intervals thereafter. At the conclusion of ten hours, the samples were opened and a fifty milliliter sample was pipetted from each layer. The fifty milliliter samples were titrated to a phenolphthalein end point with a standardized solution of alcoholic sodium hydroxide. The proper calculations were made to convert the units to pound mols per cubic feet of solution for plotting the equilibrium diagram.

The results of these determinations indicate that the data obtained is within a 10% deviation range of the published data so that the discrepancies may be accounted for in the above listed material differences. These data are listed in Table II and the plot is shown by Figure 2.

Procedure - The water supply tank was filled and the water neutralized to a methyl orange end point with concentrated hydrochloric acid. Compressed air was used as the mixing mechanism. The water was then heated by open steam to the same temperature as the toluene in the storage tanks. The pump servicing the constant head tank from the water storage tank was started and the by-pass valve was adjusted so as to regulate the flow to the constant head tank such that the overflow from this tank back to the storage tank was enough to maintain the constant head leval at the various flow rates used in the runs. The water was admitted into the top of the column through the continuous phase distributor after passing through a calibrated orifice.

The toluene-benzoic acid solution from a previous run,

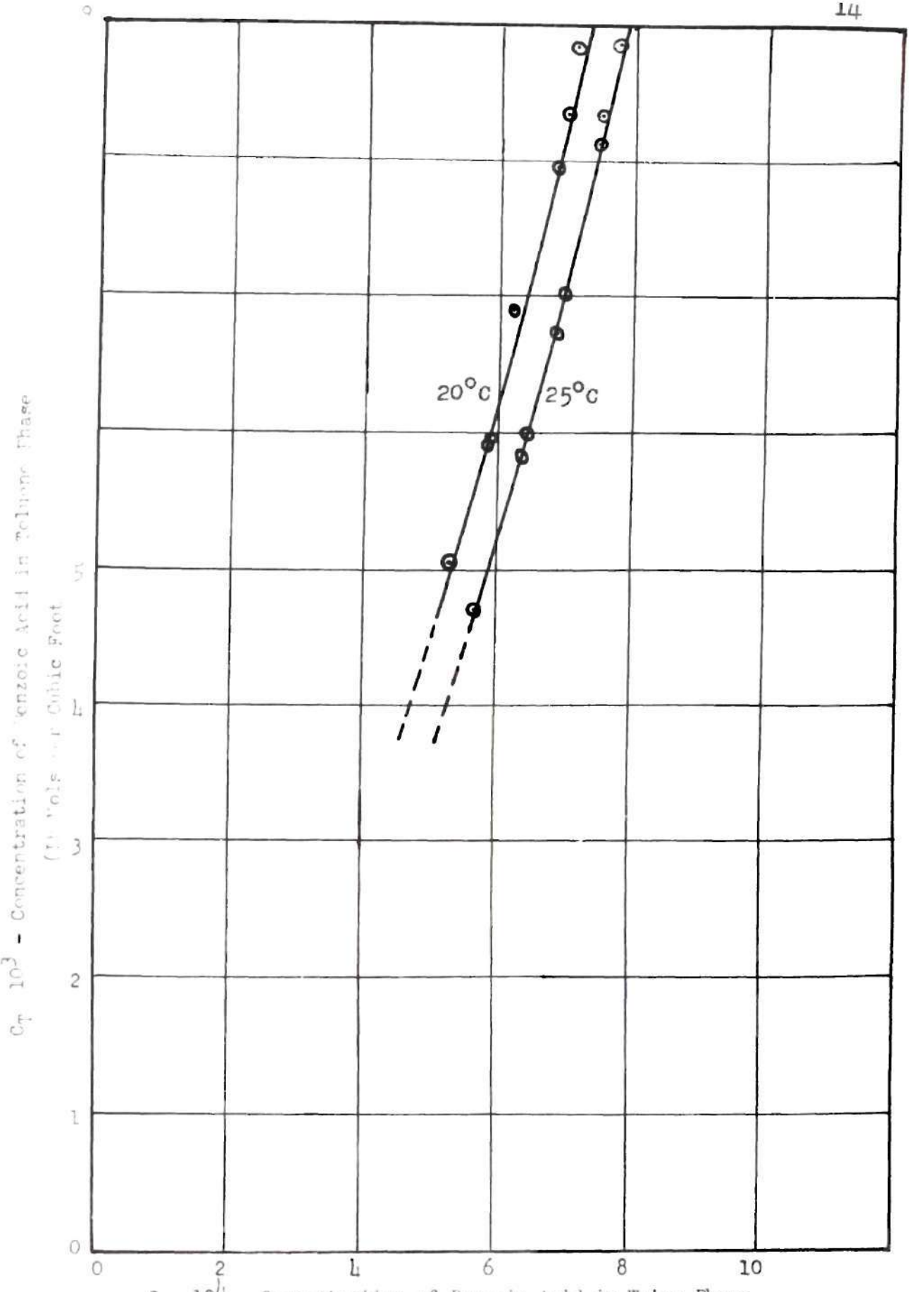

FIG. 2 EQUILIBRIUM DISTRIBUTION BENZOIC ACID-TOLUENE-WATER

contained in the toluene receiving tank was pumped back to the toluene supply tank. In this tank the concentration of benzoic acid in the toluene was brought back to the desired strength of .0006 pound mols of benzoic acid per cubic foot of toluene. The amount of benzoic acid added was determined from the amount of toluene solution used and the concentration of the exit toluene solution of the previous run. All mixing was manual in the toluene tanks. The toluene was then pumped to the constant head tank and the flow adjusted so that the overflow back to the supply tank was sufficient to maintain a constant head for the flow desired. The toluene was then admitted to the bottom of the tower through the dispersed phase distributor.

The column was filled with water to about two-thirds its height and the flow adjusted to the desired rate by the orifice manometer. The toluene was then introduced into the column and its flow was adjusted to the desired rate by its orifice manometer. The level of the interface between the two phases was then adjusted by its control standpipe. A final adjustment was then made on the flow rates. At intervals throughout this work the orifice manometer calibrations were checked by the direct weighing method. The agreement between these check points and the original calibration curve was always quite good. The column was operated from thirty to forty five minutes to allow steady state conditions to be reached. After this interval, samples of the outlet toluene

and the outlet water were taken every five minutes. Samples of the inlet toluene were taken during the first part of the run and again at the end of the run. All samples were titrated with alcoholic sodium hydroxide to a phenophthalein end point. The sodium hydroxide used was approximately 0.15 normal and was titrated against standard benzoic acid to obtain its exact normality before each run. The run was completed when four consecutive toluene samples showed a constant titration.

Phenomena such as coalescence, channeling, and flooding tendency were studied during the interval between the interface adjustment and the time steady state conditions were reached.

THEORETICAL BACKGROUND

Many of the leaders in the "diffusional" operations field have shown quite conclusively that mass transfer rate equations that apply to absorption and distillation operations may also be applied to the liquid-liquid extraction operation.

Elgin and Browning[8] developed the equation for rate of mass transfer for a liquid-liquid extraction system by starting with the equation:

$$\frac{dN_t}{A d\theta} = k_w(C_w - C_{wi}) = k_t(C_{ti} - C_t) \qquad (1)$$

which is the general equation giving the instantaneous rate of extraction from phase w to phase t in a system involving diffusion across two laminar films in series and a time of contact sufficient to permit the establishment of steady state concentration gradients across the laminar layers. They showed that for cases where the system does not depart considerably from the simple distribution law, and where volume changes, amount extracted, and concentrations involved were small, the equation may be integrated to give the convenient relation

$$N/\theta = K_w a V \qquad \frac{(C_{w1}^* - C_{w1}) - (C_{w2}^* - C_{w2})}{\log_e \dfrac{(C_{w1}^* - C_{w1})}{(C_{w2}^* - C_{w2})}} \qquad (2)$$

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8 Elgin, J. C., and Browning, F. M.; Transactions American Institute of Chemical Engineers, 31, 639 (1935); 32, 105 (1936)

Equation (3) and (4) readily follow from equation (2)

$$N/\theta = K_w a V \frac{C_1 - C_2}{2.3 \log \left(\frac{C_1}{C_2}\right)} \qquad (3)$$

$$K_w a = \frac{N/\theta}{V(\Delta C)\text{log mean}} \qquad (4)$$

In the work of Chilton and Colburn[9] the term H.T.U. was introduced in computations for distillation and absorption in packed columns. They showed the relation of the H.T.U. values to the above mass transfer coefficient and pointed out advantages of the use of the H.T.U. method over the mass transfer coefficient method.

Elgin and Browning[10] employed an analogous procedure to that of Chilton and Colburn[9] and obtained the following expressions for liquid-liquid extraction:

$$(\text{H.T.U.})_{ow} = \frac{H}{n} = \frac{H}{\int_{C_{w1}}^{C_{w2}} \frac{dC_w}{C}} = \frac{V'_w}{K_w a S} \qquad (5)$$

Since the simple distribution law valid, and the solution used is dilute; equation (5) may be simplified to

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9 Chilton, T. H., and Colburn, A. P.; Industrial Engineering Chemistry, 27, 255-60, 904 (1936)

10 Elgin, J. C., and Browning, F. M.; Transactions American Institute of Chemical Engineers, 31, 639 (1935); 32, 105 (1936)

$$(H.T.U.)_{ow} = \frac{H}{n} = \frac{H}{\frac{C_{w2} - C_{w1}}{C \text{ log mean}}} = \frac{V_w}{K_{wa}} \qquad (6)$$

Colburn[11] pointed out that many operations such as dehumidification, absorption, distillations, and extraction involved the two film resistance theory, whereupon he derived an expression relating the overall H.T.U. to single film values of H. T.U. This relation is stated as follows:

$$(H.T.U.)_{ow} = (H.T.U.)_w + (H.T.U.)_t \frac{(V_w \, dC_w)}{(V_t \, dC_t)} \qquad (7)$$

Colburn's[11] method of correlation of data follows directly from the equation (7) since this equation can easily be shown to be that of a straight line. A plot of this equation using $(H.T.U.)_{ow}$ as the ordinate and $\frac{V_w \, dC_w}{V_t \, dC_t}$ as the abscissa will result in a linear plot whose intercept is $(H.T.U.)_w$ and whose slope is $(H.T.U.)_t$. This method of correlation of data is used to advantage in this thesis.

11 Colburn, A. P.; Transactions American Institute of Chemical Engineers, 35, 211 (1939)

CALCULATIONS

The capacity coefficient $K_w a$ was calculated from equation (4) for all runs. The $(H.T.U.)_{ow}$ was in turn calculated by use of equation (6). The results of the calculations are shown in Tables III and IV of Appendix II. These results were then correlated according to equation (7) by plotting the term $\frac{V_w}{V_t}\frac{dC_w}{dC_t}$ as the abscissa verses $(H.T.U.)_{ow}$ as the ordinate. This correlation is shown by Figure 3 and Figure 4. The term $\frac{dC_w}{dC_t}$, the slope of the equilibrium curve, was computed by taking the instantaneous slope of the equilibrium curve at the average toluene concentration in the column. The actual slope measurements were made by the use of a tangentometer and the values obtained were plotted against toluene concentration for convience. This plot is shown in Figure 9 and its accompanying data tabulated in Table V. Since the differences in slopes of the equilibrium curves for 20° and 25°C. were so slight, Figure 9 may be used for either case. The slopes of the lines in Figures 3 and 4 gave the values for the $(H.T.U.)_t$ and the intercepts of these lines gave the values for the $(H.T.U.)_w$. These results are tabulated in Table VI.

A sample calculation for $(H.T.U.)_{ow}$ is made herein by using run 4C.

(1) Calculation of (N/θ)

$$(N/\theta) = (C_{t1} - C_{t2})(V_t)(S)$$

$$= (64.79 - 58.81)(10^{-4})(16.05)(0.1655)$$

$$= 15.9 \times 10^{-4} \text{ lb. mols/hr.}$$

(2) Calculation of (ΔC) log mean

$$(\Delta C) \text{ log mean} = \frac{(C^*_{w1} - C_{w1}) - (C^*_{w2} - C_{w2})}{2.3 \log \frac{(C^*_{w1} - C_{w2})}{(C^*_{w2} - C_{w2})}}$$

From Figure 2

$$C^*_{w1} = 6.6 \times 10^{-4}$$

$$C^*_{w2} = 6.3 \times 10^{-4}$$

$$= \frac{(6.60 \times 10^{-4} - 0)(6.30 \times 10^{-4} - 1.61 \times 10^{-4})}{2.3 \log \frac{(6.60-0)}{(6.30-1.61)}}$$

$$= 5.6 \times 10^{-4} \text{ lb. mols/cu. ft.}$$

(3) Calculation of K_{wa}

$$K_{wa} = \frac{N/\theta}{V(\Delta C) \text{ log mean}}$$

$$= \frac{15.9}{(0.99)(5.6 \times 10^{-4})}$$

$$= 2.86/\text{hrs.}$$

(4) Calculation of $(H.T.U.)_{ow}$ using equation (6)

$$(H.T.U.)_{ow} = \frac{V_w}{K_w a}$$

$$= \frac{59.39}{2.86}$$

$$= 20.8 \text{ ft.}$$

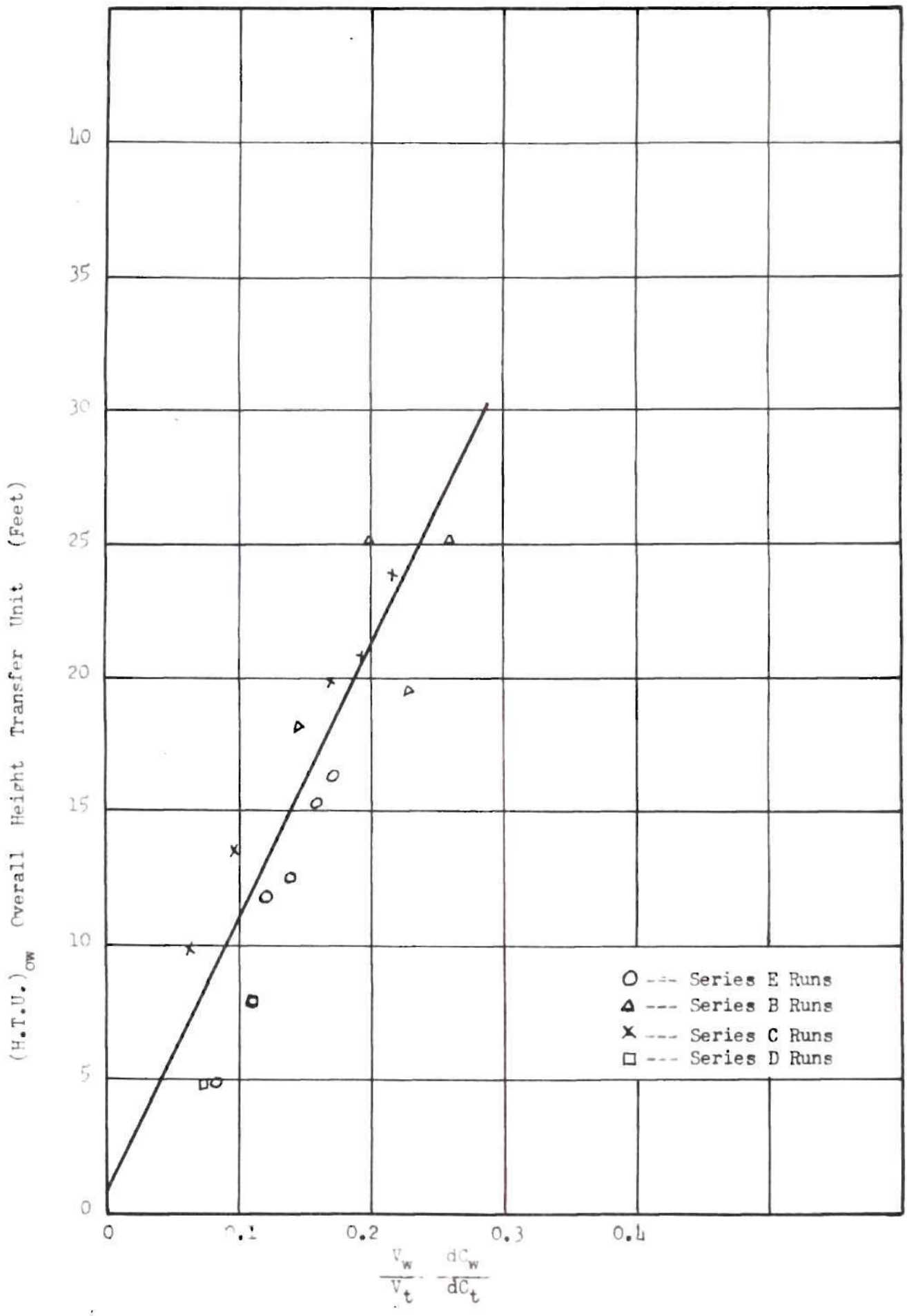

Fig. 3 $(H.T.U.)_{ow}$ Values For Fiberglas Packing

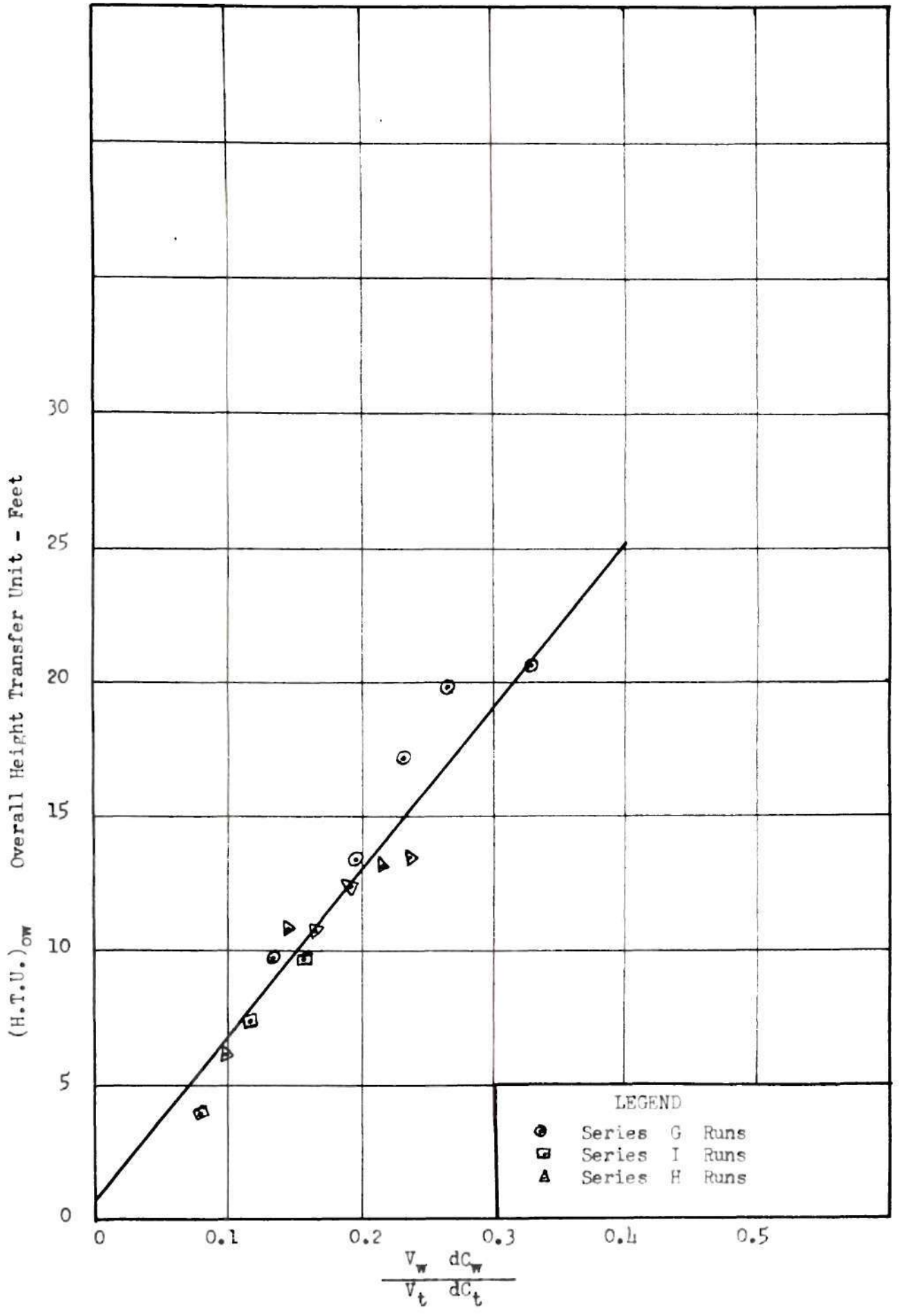

Fig. 4 $(H.T.U.)_{ow}$ VALUES FOR RASCHIG RING PACKING

RESULTS

The H.T.U. method of correlation of data proved to be a simple and practical method. The results of this work indicate that this method of correlation does not possess a satisfactory degree of accuracy, although the method is believed adequate for design work. Figures 3 and 4 will illustrate this point. This method, however, does present the performance data of the various packings in a clear and concise manner and will tend to eliminate much of the time consuming laboratory investigations found necessary in other correlation methods. Figure 5 is a plot compiling the data from Figures 3 and 4 along with recently published data* to give a more complete conception of the performances of the various packings studied. The advantages of the H.T.U. correlation method is clearly brought out in Figure 5. As an example, with other conditions being equal the performance superiority of Raschig Rings as a packing over Fiberglas is clearly shown. This does not mean, however, that Raschig Rings are the superior packing in every problem. Figure 5 gives no indication of such variables as weight per packed foot, pressure drop, holdup, and packing life. These

*Curves No. 1, 3, 5, 6 of Figure 8 are taken from an article in Transactions American Institute of Chemical Engineers, 37, 559 (1941) by S. B. Row, J. H. Koffolt, and J. R. Withrow entitled "Characteristics and Performance of a Nine Inch Liquid-Liquid Extraction Column."

variables must also be carefully considered when choosing a packing material. Another advantage in this method is that each curve represents different toluol and water flow rate ratios.

The simple and practical feature of the H.T.U. method should be readily apparent from an inspection of Figure 5 For instance, if a designer had a certain separation to make in a liquid-liquid extraction tower; that is, the V_t was fixed, he could assume a V_w and calculate the numerical value of the abscissa of Figure 5. He can then readily see the different tower heights necessary in the use of the various packings. Now he would be able to chose his packing from an overall economic balance on the various packings. The process would then be repeated using different V_w values until a minimum in the overall cost was obtained.

Runs 5B, 6B, 3D, and 6E were omitted from the plot in Figure 7 since they were by observation far removed from the great majority of points. These errors can for the most part, be accounted for in the fact that the system was not at equilibrium when the final readings were taken. Runs 5I, 6I, and complete J series were omitted from the correlation in Figure 4 since by visual observations the column was known to be flooding during these runs. This flooding presented itself in the form of violent frothing and toluol build-up just under the packed section.

Deviations of Figures 3 and 4 from straight line relations is believed to occur at higher water rates as would be indicated by Figures 6 and 7. Both of these plots in which $(H.T.U.)_{ow}$ is plotted against water flow rates has a very definite curvature at the higher rates. Up to flow rates of sixty cubic feet per hour per square foot of column area these curves show very little tendency to vary from a linear function; however, at this point there occurs a marked break in the curves. As the toluol rate is increased, this break point seems to occur at lower water rates. All indications are that above these breaking points the H.T.U. method of correlation would not be valid. These deviations would indicate that factors other than flow rates affect the H.T.U. value. Beilder[12] working in conjunction with Row, Koffolt, and Withrow[13] has shown photographically that high toluene and water flow rate resulted in a breaking down of the liquid bubbles into smaller ones, resulting in a greater extraction rate, therefore, lower overall H.T.U. values.

The plot of $(H.T.U.)_{ow}$ versus the dispersed phase flow rate in cubic feet per hour per square foot of column area

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12 Beidler, E.A., Coffolt, J. H., and Withrow, J. R.; Preliminary Investigation Mechanism Liquid-Liquid Extraction. AD motion picture study.

13 Row, S. B., Koffolt, J. H., and Withrow, J. R.; Transactions American Institute of Chemical Engineers, 37, 559 (1941)

shown in Figure 8 lends itself in support of the H.T.U. correlation method and throughout the flows investigated shows no marked deviation from its presumed behavior.

Of the two packings compared the one-half inch Raschig Rings proved to have better performance characteristics than the Fiberglas tower packing. As shown in Figure 5, the Fiberglas packing has a performance characteristic somewhat better than the spray and bubble cap columns but it does not compare favorably with Raschig Ring and Berl Saddle packing.

The correlation curve acquired from runs made with Raschig Ring packing check fairly accurately the previously published data on this type packing. See Figure 5 for this comparison.

The flooding velocity of the Fiberglas packing is very high and was not reached in this investigation. The Raschig Ring packing exhibited a lower flooding velocity. This velocity was exceeded for toluol flows of 19.6 when water flows were greater than 67.4. All toluol flows greater 19.6 flooded the column regardless of the water flow. The above flows are all in units of cubic feet per hour per square foot of column area.

The amount of coalescense in the two types of packings used was about the same. Figure 13 of Appendix IV is a photograph of the bottom and top distributors made during a Fiberglas run showing to some extent the coalescense present. The interface level has been raised above its normal position for the

purpose of this photograph.

Channeling was present to some extent with both packings.

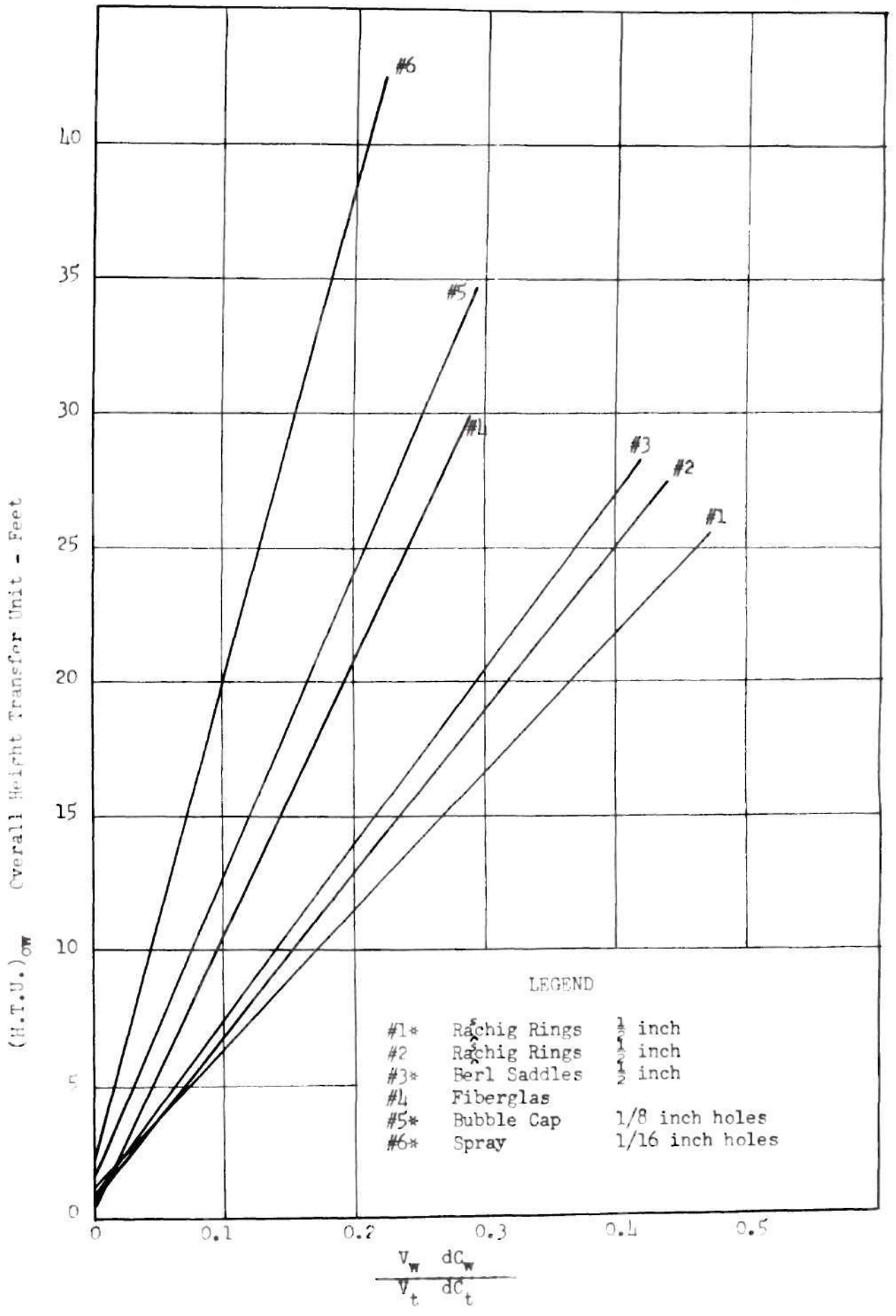

Fig. 5 COMPARISON OF $(H.T.U.)_{ow}$ VALUES

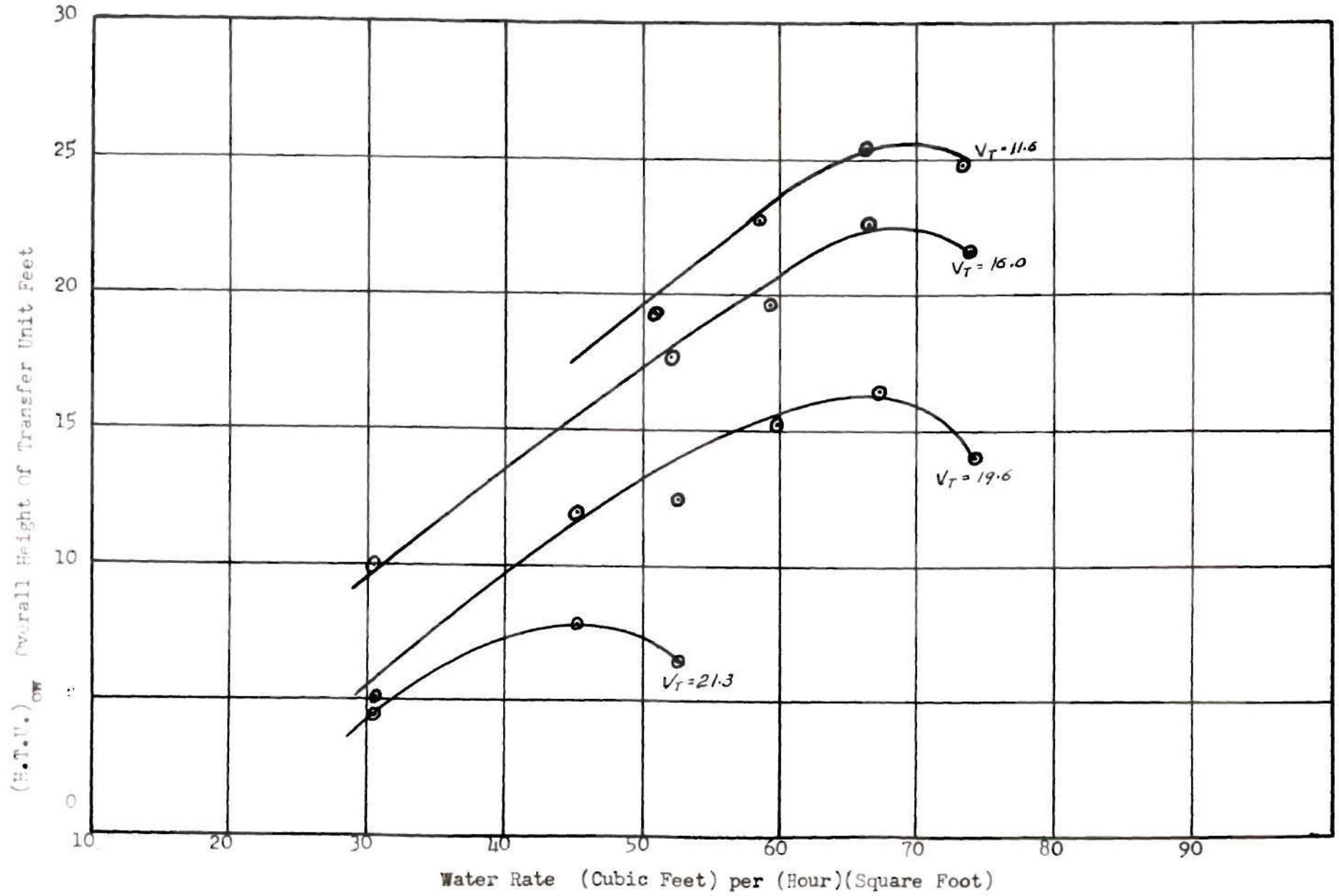

Fig. 6 COMPARASION OF $(H.T.U.)_{ow}$ VALUES WITH VARYING TOLUENE AND WATER RATES. FIBERGLAS PACKING

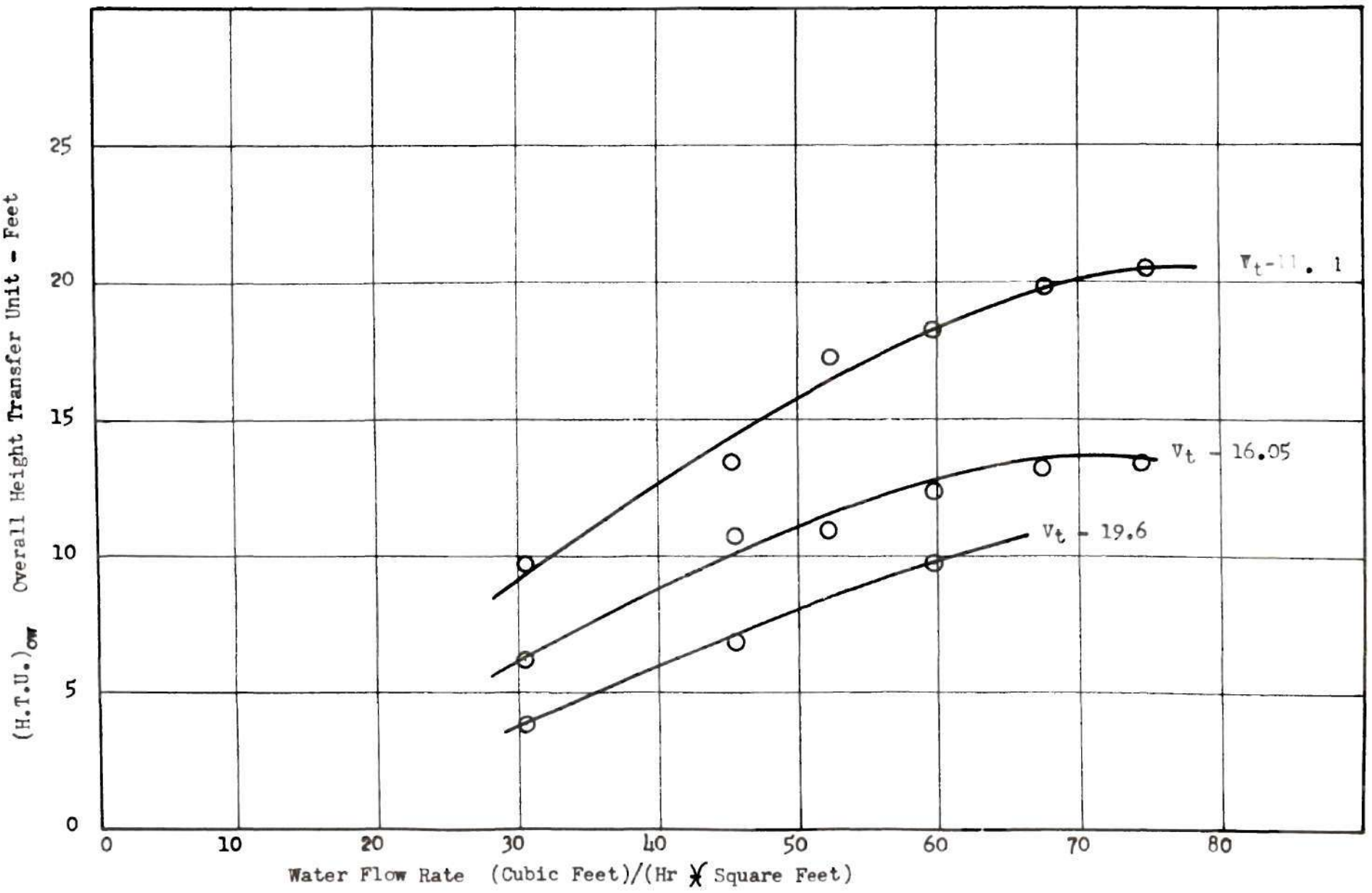

Fig. 7 COMPARISON OF $(H.T.U.)_{ow}$ VALUES VARYING TOLUENE AND WATER RATES. RASCHIG RING PACKING

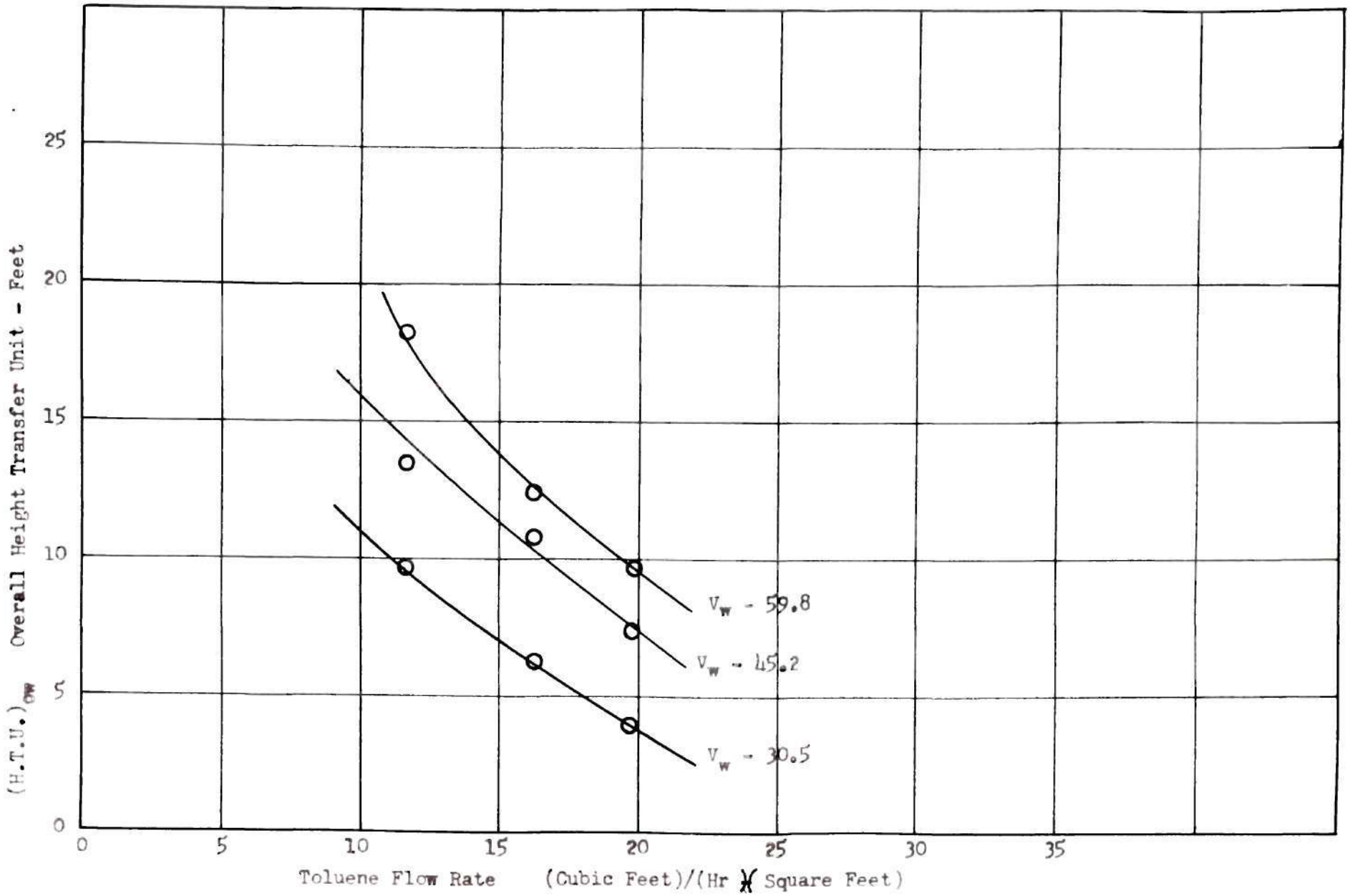

Fig. 8 COMPARISON OF (H.T.U.)$_{ow}$ VALUES VARYING TOLUENE AND WATER RATES. RASCHIG RING PACKING

SUMMARY

In this work two types of packing materials were investigated under a wide range of flow conditions, involving somewhat over fifty runs. The two packings investigated were Fiberglas and one-half inch unglazed chemical stoneware Raschig Rings. The results of this work indicated:

(1) Correlation of data was greatly facilitated by the use of Colburn's correlation equation for which the overall $(H.T.U.)_{ow}$ was plotted against the term $\frac{V_w \, dC_w}{VT \, dC_t}$ to give a straight line relation.

(2) The slope of the straight line which is the $(H.T.U.)_t$ (height of transfer unit, toluene film) was found to be the decisive factor in indicating the performance of various packings used.

(3) Plots of the $(H.T.U.)_{ow}$ against water flows was found to deviate somewhat from a linear function when the water flow became large. This fact would question the validity of the Colburn equation when the ration V_w/V_t becomes large.

(4) One-half inch stoneware Raschig Rings were found to be the more efficient of the two packings investigated.

(5) The results of the runs made on the Raschig Rings checked within experimental limits the results of similar runs found in the literature. Appendix I carries the complete table of nomenclature used in this report.

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APPENDIX I

Table of Nomenclature

A	Total surface area between phases, sq. ft.
C_t	Concentration of benzoic acid in toluene, lb.mols/cu.ft.
C_w	Concentration of benzoic acid in water, lb.mols/cu.ft.
C_w^*	Concentration of benzoic acid in water which would be in equilibrium with concentration, C_t, in toluene phase, lb.mols/cu.ft.
d	Differential operator
$\frac{dC_w}{dC_t}$	Slope of equilibrium distribution curve
H	Effective height of extraction column, feet
H.T.U.	Height of one transfer unit, feet
k	Individual film transfer coefficient
$K_w a$	Overall coefficient of mass transfer
N/θ	Pound mols of benzoic acid transferred in unit time, lb.mols/hr.
n	Number of transfer units
S	Cross-section of column, sq.ft.
V	Effective volume of column, cu.ft.
V_w	Superficial velocity water phase, cu.ft./(Hr.) (sq.ft.)
V_w'	Rate of flow of water phase, cu.ft./hr.
V_t	Superficial velocity toluene phase, cu.ft./(hr.) (sq.ft.)
V_t'	Rate of flow of toluene phase, cu.ft./hr.

APPENDIX I

Table of Nomenclature (Cont'd.)

Subscripts

1 and 2	Terminal conditions
i	At interface
O	Overall
t	Toluene or toluene film
w	Water or water film

APPENDIX II

TABLE II: Determination of Equilibrium Distribution

Run No.	Temp. °C	Concentrations Lb. Mol. Benzoic/Cu.Ft. Water Layer	Toluene Layer
1	20	.000685	.00792
2	20	.000620	.00689
3	20	.000594	.00599
4	20	.000707	.00882
5	20	.000700	.00837
6	20	.000620	.00664
7	20	.000589	.00593
8	25	.000774	.00880
9	25	.000744	.00835
10	25	.000698	.00702
11	25	.000640	.00600
12	25	.000744	.00814
13	25	.000682	.00678
14	25	.000635	.00587

APPENDIX II

TABLE III: Summary of Data and Calculated Results

Run No.	Run Temp °C	V_w	V_t	C_{t1} x10^{-4}	C_{t2} x10^{-4}	C_{w1} x10^{-4}	K_wa	$(H.T.U.)_{ow}$
1 B	25	26.66	9.23	66.85	60.93	2.05	1.46	18.2
2 B	25	44.85	10.79	66.03	60.49	1.33	1.49	30.1
3 B	25	50.91	11.61	66.55	58.01	1.95	2.66	19.5
4 B	23	58.30	11.61	67.34	59.89	1.48	2.25	25.2
5 B	23	66.06	11.61	67.29	59.01	1.46	2.49	25.4
6 B	25	73.03	11.61	66.32	57.18	1.53	2.88	24.8
7 B	25	43.94	11.61	66.42	60.47	1.52	1.77	25.3
1 C	20	30.48	16.05	65.53	60.34	2.72	3.09	13.5
2 C	20	20.00	16.05	65.53	62.38	2.53	1.48	9.9
3 C	24	52.12	16.05	65.21	59.57	1.73	2.72	19.2
4 C	24	59.39	16.05	64.79	58.81	1.61	2.86	20.8
5 C	25	66.66	16.05	64.20	58.22	1.44	2.81	23.7
6 C	25	73.64	16.05	64.69	57.79	1.50	3.24	22.8
1 D	25	30.48	21.32	66.64	59.94	4.69	6.50	4.8
2 D	25	45.21	21.32	66.23	59.02	3.40	5.58	7.7
3 D	25	52.42	21.32	68.88	58.78	4.10	8.61	6.1
1 E	25	30.48	19.61	64.20	57.19	4.51	6.22	4.9
2 E	25	45.21	19.61	64.99	59.06	2.57	3.79	11.9
3 E	25	52.42	19.61	64.97	58.37	2.48	4.22	12.4
4 E	25	59.80	19.61	65.48	58.96	2.14	3.96	15.1
5 E	25	67.39	19.61	65.28	58.47	1.98	4.09	16.5
6 E	25	74.39	19.61	64.44	55.93	2.24	5.34	13.9
1 G	22	30.48	11.61	73.23	65.31	3.02	3.16	9.6
2 G	22	45.21	11.61	73.18	64.37	2.33	3.40	13.3
3 G	22	52.42	11.61	73.35	64.73	1.90	3.04	17.2
4 G	24	59.80	11.61	70.25	60.71	1.85	3.29	18.2
5 G	24	67.39	11.61	70.25	60.32	1.71	3.38	19.9
6 G	25	74.39	11.61	71.34	60.52	1.69	3.62	20.6
1 H	22	30.48	16.05	73.30	65.55	4.08	5.05	6.1
2 H	25	45.21	16.05	71.41	63.24	2.90	4.21	10.7
3 H	24	52.42	16.05	70.31	61.01	2.84	4.93	10.6
4 H	24	59.80	16.05	71.17	61.67	2.55	4.85	12.3
5 H	24	67.39	16.05	71.09	60.89	2.43	5.18	13.0
6 H	24	74.39	16.05	71.09	60.31	2.33	5.54	13.4

TABLE III (Continued)

Run No.	Run Temp °C	V_w	V_t	C_{t1} x10^{-4}	C_{t2} x10^{-4}	C_{w2} x10^{-4}	$K_w a$	$(H.T.U.)_{ow}$
1 I	24	30.48	19.61	70.25	62.27	5.13	7.71	4.0
2 I	25	45.21	19.61	71.49	62.85	3.75	6.15	7.4
3 I	Data no good on this run							
4 I	25	59.80	19.61	70.11	60.74	3.08	6.20	9.7
5 I	22	67.39	19.61	70.98	64.00	2.03	4.30	15.7
6 I	22	74.39	19.61	70.98	63.61	1.94	3.66	20.3
1 J	24	30.48	21.32	70.25	64.21	4.21	5.09	6.0
2 J	25	45.21	21.32	71.15	62.86	3.91	6.58	6.9
3 J	22	52.42	21.32	70.98	64.00	2.84	5.12	10.2
4 J	22	59.80	21.32	70.98	64.00	2.49	4.91	12.2
5 J	22	67.39	21.32	70.98	63.80	2.28	4.92	13.7

APPENDIX II

TABLE IV: Calculated Data for Correlation

Run	V_w	V_t	(C_t)Ave	$\frac{dC_w}{dC_t}$	$\frac{V_w dC_w}{V_t dC_t}$	H.T.U.
1 B	26.66	9.23	6.389	.0520	.147	18.2
2 B	44.85	10.79	6.326	.0522	.217	30.1
3 B	50.91	11.61	6.228	.0524	.229	19.5
4 B	58.30	11.61	6.362	.0521	.261	25.2
5 B*	66.06	11.61	6.365	.0521	.297	25.4
6 B*	73.03	11.61	6.175	.0525	.330	24.8
7 B	43.94	11.61	6.359	.0521	.197	
1 C	30.48	16.05	6.294	.0523	.099	13.5
2 C	20.00	16.05	6.406	.0522	.065	9.9
3 C	52.12	16.05	6.239	.0524	.170	19.2
4 C	59.39	16.05	6.280	.0523	.193	20.8
5 C	66.66	16.05	6.121	.0527	.219	23.7
6 C	73.64	16.05	6.124	.0525	.237	22.8
1 D	30.48	21.32	6.329	.0522	.075	4.8
2 D	45.21	21.32	6.262	.0523	.111	7.7
3 D*	52.42	21.32	6.383	.0521	.128	6.1
1 E	30.48	19.61	6.069	.0527	.082	4.9
2 E	45.21	19.61	6.203	.0524	.121	11.9
3 E	52.42	19.61	6.168	.0525	.140	12.4
4 E	59.80	19.61	6.222	.0523	.159	15.1
5 E	67.39	19.61	6.238	.0522	.172	16.5
6 E*	74.39	19.61	6.019	.0528	.200	13.9
1 G	30.48	11.61	6.877	.0513	.135	9.6
2 G	45.21	11.61	6.877	.0513	.200	13.30
3 G	52.42	11.61	6.904	.0512	.231	17.2
4 G	59.80	11.61	6.548	.0517	.266	18.2
5 G	67.39	11.61	6.528	.0516	.300	19.9
6 G	74.39	11.61	6.593	.0515	.330	20.6
1 H	30.48	16.05	6.943	.0511	.097	6.1
2 H	45.21	16.05	6.733	.0513	.145	10.7
3 H	52.42	16.05	6.566	.0516	.169	10.6
4 H	59.80	16.05	6.642	.0514	.192	12.3
5 H	67.39	16.05	6.599	.0515	.216	13.0
6 H	74.39	16.05	6.570	.0515	.2391	13.4

*Runs not used in plotting Figure 5.

TABLE IV (Continued)

Run	V_w	V_t	(C_t)Ave	$\frac{dC_w}{dC_t}$	$\frac{V_w dC_w}{V_t dC_t}$	H.T.U.
1 I	30.48	19.61	6.626	.0514	.0799	4.0
2 I	45.21	19.61	6.717	.0513	.1183	7.4
3 I	Data no good on this run					
4 I	59.80	19.61	6.543	.0516	.1574	9.7
5 I	67.39	19.61	6.749	.0513	.1763	15.7*
6 I	74.39	19.61	6.714	.0514	.1954	20.3*
1 J	30.48	21.32	6.823	.0512	.0732	6.0*
2 J	45.21	21.32	6.701	.0513	.1088	6.9*
3 J	52.42	21.32	6.749	.0512	.1259	10.2*
4 J	59.80	21.32	6.749	.0512	.1436	12.2*
5 J	67.39	21.32	6.739	.0512	.1618	13.7*

*Column Flooding - not plotted

APPENDIX II

TABLE V: Data for Derivative Curve

Average Toluene Concentration (Lb Mols Benzoic Acid/Cu.Ft.Toluene)	Derivative dC_w/dC_t
0.50	0.0567
0.55	0.0552
0.60	0.0527
0.65	0.0518
0.70	0.0510
0.75	0.0505
0.80	0.0498
0.85	0.0502

TABLE VI: Tabulation of Individual Phase H.T.U. Values

Packing	Range of Flow Cu.Ft./(Hr.)(Sq.Ft.) Water	Toluene	$(H.T.U.)_w$ Ft.	$(H.T.U.)_t$ Ft.
½" Raschig Rings	30.5 - 74.4	11.6 - 19.6	0.95	60
Fiberglas	30.5 - 74.4	11.6 - 19.6	1.00	101

APPENDIX II

TABLE VII: Calibration Data

Water Orifice

Manometer Reading Inches	Flow Rate Cu.Ft./Hr.	Manometer Reading Inches	Flow Rate Cu.Ft./Hr.
0.2	1.861	4.8	10.910
0.4	2.992	5.1	11.231
0.6	3.674	5.2	11.311
0.8	4.372	5.4	11.632
1.0	5.030	5.7	11.873
1.2	5.254	5.8	12.193
1.4	5.840	6.0	12.274
1.6	6.257	6.3	12.595
1.8	6.738	6.4	12.755
2.0	7.460	7.1	13.397
2.3	7.701	7.5	13.798
2.5	7.942	7.7	13.958
2.8	8.423	8.0	14.199
3.1	8.664	8.3	14.440
3.4	9.145	8.9	15.001
3.5	9.225	9.3	15.402
3.8	9.626	9.7	15.803
4.0	9.867	10.2	16.044
4.5	10.509		

APPENDIX II

TABLE VIII: Calibration Data

Toluene Orifice

Manometer Reading Inches	Flow Rate Cu.Ft./Hr.	Manometer Reading Inches	Flow Rate Cu.Ft./Hr.
0.2	1.523	3.6	4.661
0.4	1.781	3.8	4.809
0.6	2.049	4.0	4.892
1.0	2.649	4.2	5.030
1.2	2.834	4.4	5.123
1.4	3.018	4.8	5.353
1.6	3.203	5.0	5.473
1.8	3.369	5.4	5.686
2.0	3.518	5.7	5.473
2.2	3.674	6.0	5.926
2.4	3.849	6.5	6.184
2.6	3.997	6.7	7.218
2.8	4.126	7.0	6.479
3.0	4.292	7.3	6.590
3.2	4.421	7.5	6.673
3.4	4.495	8.0	6.923

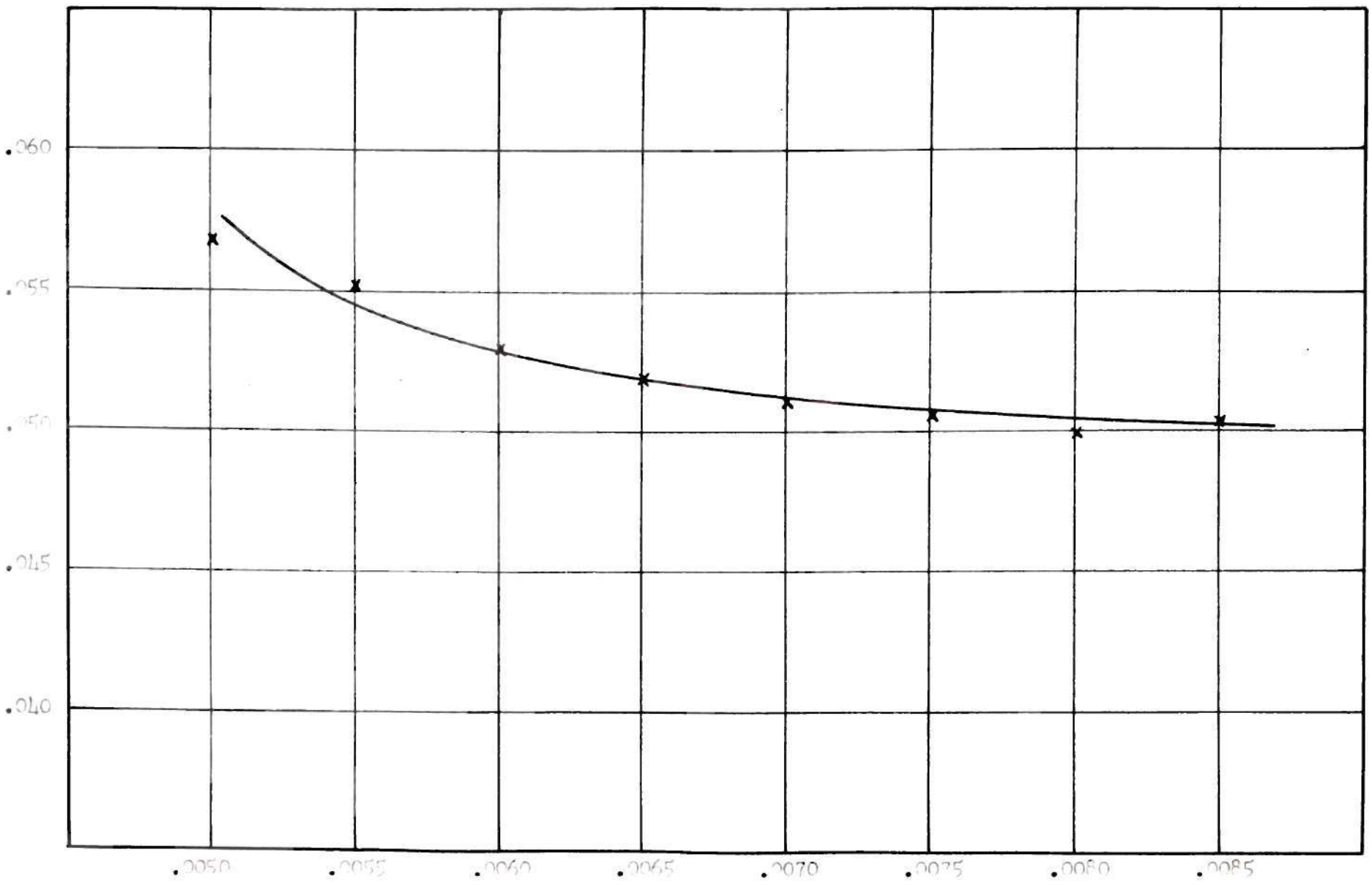

Fig. 9 DERIVATIVES OF EQUILIBRIUM DISTRIBUTION CURVE

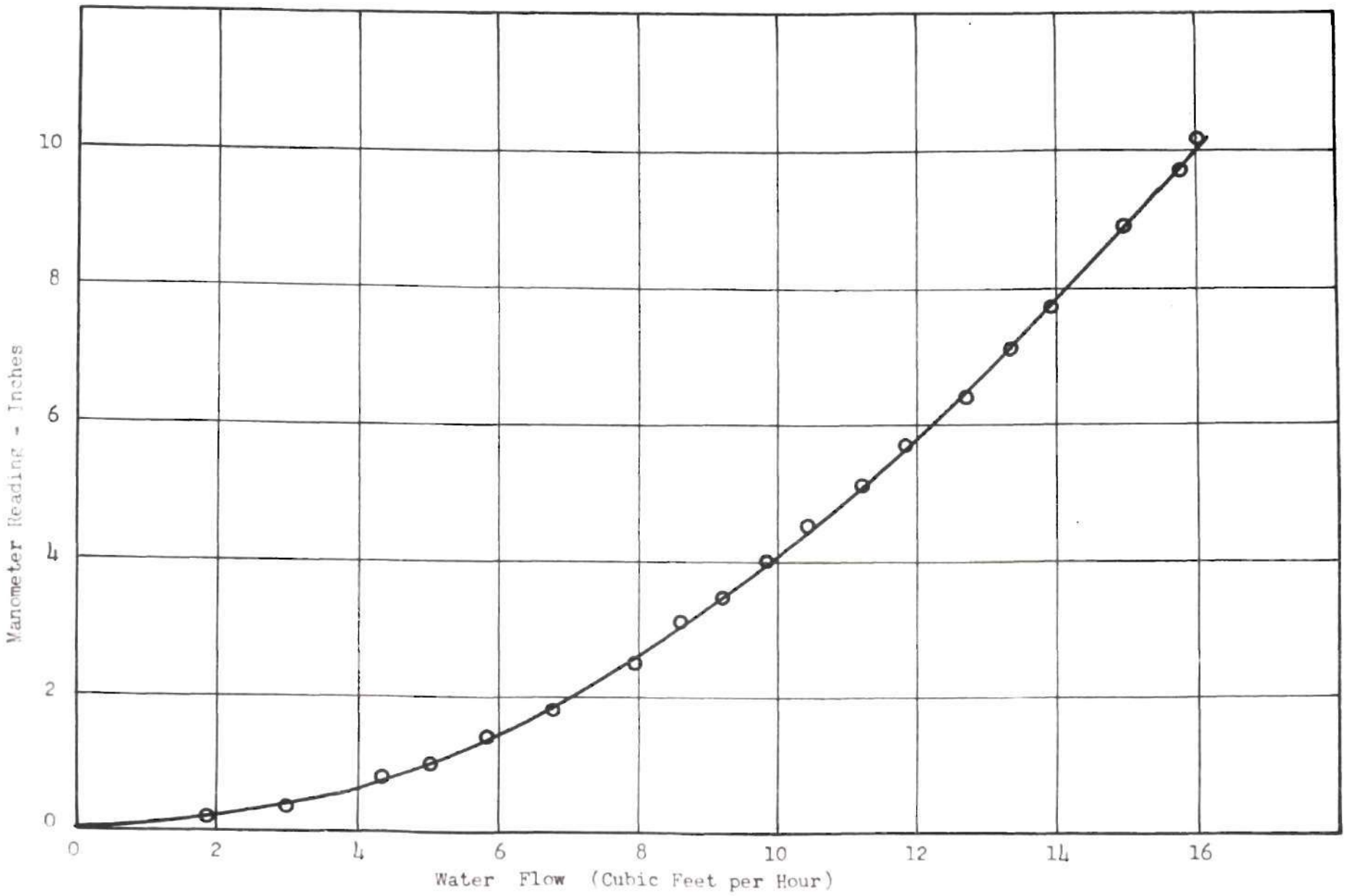

Fig. 10 WATER ORIFICE CALIBRATION CURVE

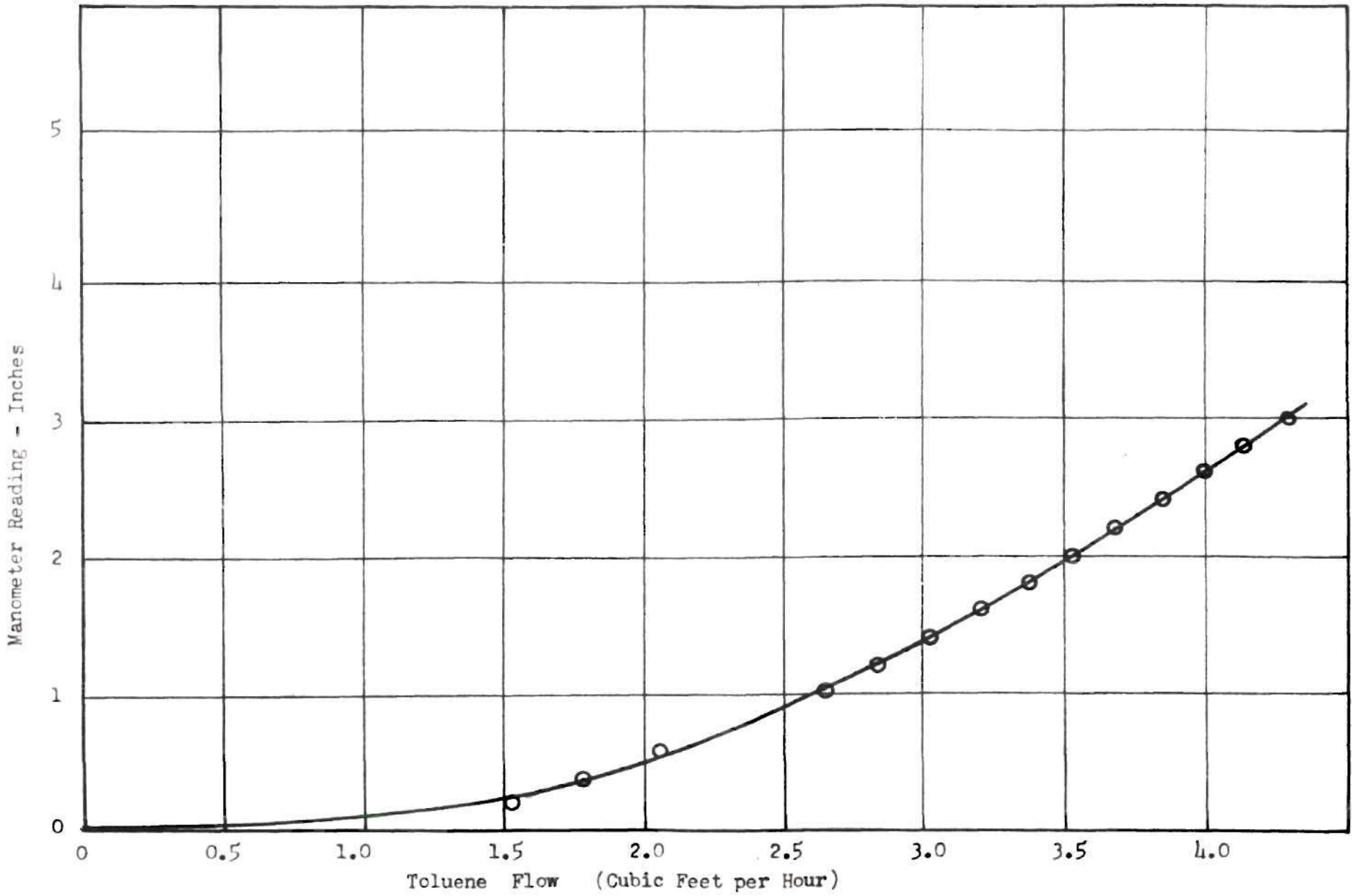

Fig. 11 TOLUENE ORIFICE CALIBRATION CURVE

APPENDIX IV

FIGURE 12 OVERALL VIEW OF EXTRACTION SYSTEM

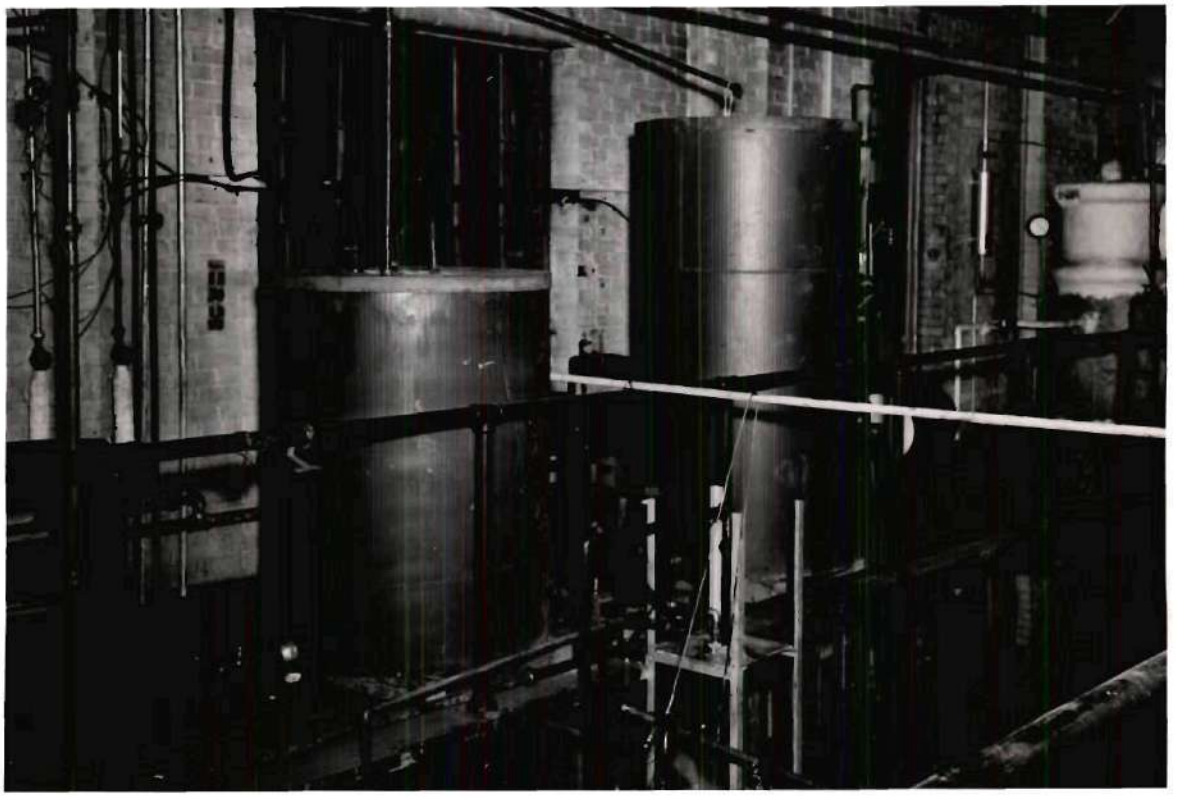

FIGURE 13 WATER AND TOLUENE SUPPLY TANKS

CONTINUOUS

DISPERSED

FIGURE 14 DISTRIBUTORS, DISPERSED AND CONTINUOUS PHASE

FIGURE 15 FIBERGLAS PACKING, AS RECEIVED